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FILING DATE: April 23, 2003

RELATED PCT APPLICATION NUMBER: PCT/US04/12828

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60/464868-042303

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Provisional Application Cover Sheet

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60/464868

This is a request for filing a PROVISIONAL APPLICATION under 37 CFR 1.53 (b)(2).

INVENTOR(s)/APPLICANT(s)		
Last Name	First Name, MI	Residence (City and Either State or Foreign Country)
Olson	Edwin S.	Grand Forks, ND
Holmes	Michael J.	Grand Forks, ND
Pavlish	John H.	East Grand Forks, MN

TITLE OF THE INVENTION  
PROCESS FOR REGENERATING A SPENT SORBENT

CORRESPONDENCE ADDRESS

Customer Number 30163



30163

PATENT TRADEMARK OFFICE

ENCLOSED APPLICATION PARTS (check all that apply)

<input checked="" type="checkbox"/>	Specification	Number of Pages: 10 (including cover page)	( X )	Small Entity Status Claimed
<input checked="" type="checkbox"/>	Drawing(s)	Number of Pages: 1 (included in the Specification)		
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By \_\_\_\_\_  
Typed Name: Bruce A. Johnson

Respectfully submitted,

Bruce A. Johnson  
Attorney for Applicant(s)  
Reg. No. 37361

Date: 4-23-03

Telephone No.: 512-301-9900

60464868 . 042303

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**Complete if Known**

Application Number	TBD
Filing Date	4/23/2003
First Named Inventor	Edwin S. Olson
Examiner Name	
Group Art Unit	
Attorney Docket No.	EER.P0018P

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- Applicant claims small entity status.
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- See 37 CFR 1.27

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101	750	201	375	Utility filing fee	
106	330	208	165	Design filing fee	
107	490	207	245	Plant filing fee	
108	750	208	375	Reissue filing fee	
114	160	214	80	Provisional filing fee	80

**SUBTOTAL (1)** \$ 80.00**2. EXTRA CLAIM FEES**

Total Claims	Extra Claims	Fee from below	Fee Paid
0	-20**= 0	X \$18	= \$0.00
Independent Claims	0	-3**= 0	X \$84 = \$0.00

**Multiple Dependent**

Large Fee Code	Entity Fee (\$)	Small Fee Code	Entity Fee (\$)	Fee Description
103	18	203	9	Claims in excess of 20
102	84	202	42	Independent claims in excess of 3
104	270	204	135	Multiple dependent claim, if not paid
109	80	209	40	** Reissue independent claims over original patent
110	18	210	9	** Reissue claims in excess of 20 and over original patent

**SUBTOTAL (2)** \$0.00**3. ADDITIONAL FEES**

Large Fee Code	Entity Fee (\$)	Small Fee Code	Entity Fee (\$)	Fee Description	Fee Paid
105	130	205	65	Surcharge - late filing fee or oath	\$0
127	50	227	25	Surcharge - late provisional filing fee or cover sheet	\$0
139	130	139	130	Non-English specification	\$0
147	2,520	147	2,520	For filing a request for <i>ex parte</i> reexamination	\$0
112	920*	112	920*	Requesting publication of SIR prior to Examiner action	\$0
113	1,840*	113	1,840*	Requesting publication of SIR after Examiner action	\$0
115	110	215	55	Extension for reply within first month	\$0
116	390	216	195	Extension for reply within second month	\$0
117	890	217	446	Extension for reply within third month	\$0
118	1,390	218	695	Extension for reply within fourth month	\$0
128	1,890	228	945	Extension for reply within fifth month	\$0
119	310	219	155	Notice of Appeal	\$0
120	310	220	155	Filing a brief in support of an appeal	\$0
121	270	221	135	Request for oral hearing	\$0
138	1,510	138	1,510	Petition to Institute a public use proceeding	\$0
140	110	240	55	Petition to revive - unavoidable	\$0
141	1,240	241	620	Petition to revive - unintentional	\$0
142	1,240	242	620	Utility issue fee (or reissue)	\$0
143	440	243	220	Design issue fee	\$0
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122	130	122	130	Petitions to the Commissioner	\$0
123	50	123	50	Petitions related to provisional applications	\$0
126	240	126	240	Submission of Information Disclosure Stmt	\$0
581	40	581	40	Recording each patent assignment per property (times number of properties)	\$0
146	710	246	355	Filing a submission after final rejection (37 CFR 1.129(a))	\$0
149	710	249	355	For each additional invention to be examined (37 CFR 1.129(h))	\$0
179	710	279	355	Request for Continued Examination (RCE)	\$0
169	900	169	900	Request for expedited examination of a design application	\$0

Other fee (specify)

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**SUBTOTAL (3)**

\$0.00

**SUBMITTED BY**

Name (Print/Type)

Bruce A. Johnson

Registration No.  
(Attorney/Agent)

37361

Telephone

512-301-9900

Signature

Date

4/23/03

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**NONPUBLICATION REQUEST  
UNDER  
35 U.S.C. 122(b)(2)(B)(i)***First Inventor*

Edwin S. Olson

*Title*PROCESS FOR REGENERATING A SPENT  
SORBENT*Attorney Docket No.*

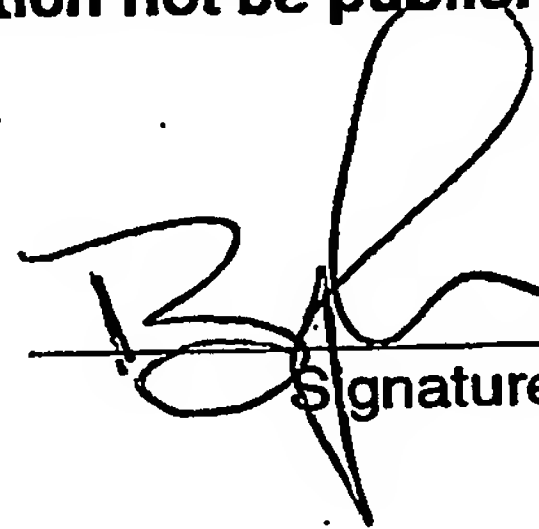
EER.P0018P

I hereby certify that the invention disclosed in the attached application has not and will not be the subject of an application filed in another country, or under a multilateral agreement, that requires publication at eighteen months after filing.

I hereby request that the attached application not be published under 35 U.S.C. 122(b).

April 23, 2003

Date



Signature

Bruce A. Johnson

Name

This request must be signed in compliance with 37 CFR 1.33(b) and submitted with the application upon filing.

Applicant may rescind this nonpublication request at any time. If applicant rescinds a request that an application not be published under 35 U.S.C. 122(b), the application will be scheduled for publication at eighteen months from the earliest claimed filing date for which a benefit is claimed.

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United States Provisional Patent Application for

**PROCESS FOR REGENERATING  
A SPENT SORBENT**

**Inventors:**

Edwin S. Olson  
Michael J. Holmes  
John H. Pavlish

**Abstract.**

A process is described for regenerating a sorbent that consists of either an injected powder material or a type of fixed or moving bed material that has been poisoned by components derived from flue gas. The sorbent is treated with an agent to remove the poisoning components and introduce a promoting agent into the sorbent.

**Outline of Examples of the Invention**

1. A process for regenerating sorbents, includes the step of washing the sorbents with a solution. The solution may be an inorganic acid solution selected from the following exemplary, non-exclusive group:  
HX (HCl, HBr, HI, HF), HNO<sub>3</sub>, HBF<sub>4</sub>, HClO<sub>4</sub>, HClO<sub>x</sub>, etc.
2. The solution may also be a salt solution selected from the following exemplary, non-exclusive group:  
MeX.....(Me = Na, K, Fe, Al, B, Zn, Mn, Mg, Ca, etc) X= halide, perchlorate, nitrate, etc.
3. The solution may also be an organic acid selected from the following exemplary, non-exclusive group:  
HX is citric, tartaric, oxalic, malonic, maleic, formic, acetic acids, etc.
4. The solution may also be an organic salt solution selected from the following exemplary, non-exclusive group:

MeX, (Me = Na, K, Fe, Al, B, Zn, Mn, Mg, Ca, etc.) X = citrate, tartrate, oxalate, malonate, maleate, formate, acetate, etc.

5. The process outlined in paragraph 1 above may include the step of selecting the sorbent from a group including powder sorbents recovered from a collection device located downstream of an injection device and bed materials removed from a sorbent bed, either sorbent type having been at least partially deactivated by the flue gas stream.
6. The process outlined in paragraph 1 above may include the step of washing the recovered powdered sorbent or the bed material in a solution of the contaminant removal agent.
7. The process outlined in paragraph 6 above may include the use of removal agents such as by washing with acid reagents.
8. The process outlined in paragraph 6 above may include the step of washing with reagents that will exchange the flue-gas derived sulfate ions and other contaminants from the binding sites on the sorbent.
9. The process outlined in paragraph 6 above may include the impregnation of agents into the sorbent to promote sorption activity. This step may be accomplished by the same reagents as listed in paragraphs 6-8 above and includes promoters such as a protonic acid HX or Lewis acid MeXn..
10. In the process outlined in paragraph 9 above, the reagents promote metal oxidation activity by creating cationic character on the carbon edge structure that attracts mercury atoms and extracts electrons from said mercury atom. A similar mechanism applies to oxidation and capture of reduced arsenic and other pollutant species.
11. The process outlined above may also use a reagent that promotes oxidation via chlorination of a sorbent surface, such as SOCl<sub>2</sub>, SO<sub>2</sub>Cl<sub>2</sub>, PCl<sub>3</sub>, POCl<sub>3</sub>, HOCl, NaOCl, Cl<sub>2</sub>, and corresponding bromine (or other halide) reagents. These can be liquid, vapor or solution reagents, depending on their stability.
12. The process outlined in paragraph 11 above may include the step of, after the washing process, dewatering and drying the sorbent.
13. In the process outlined above, the water produced in the dewatering step may be treated to remove the exchanged and solubilized contaminant species, such as sulfate, borate, phosphate, arsenate, or other oxyacid produced from flue gas or oxidation of flue gas.
14. In one example, a portion of the water removed as in the process outlined in paragraph 16 is treated in a settling basin with a Ca or Mg salt to produce insoluble Ca or Mg SO<sub>4</sub>, or other insoluble salt of the contaminant, which is removed as a sludge or solid.

- 14a. In one example, the Ca or Mg salt is lime.  
14b. In another example, the Ca or Mg salt is halide.  
14c. In another example, the Ca or Mg salt is spent solution from the resin regeneration step.

15 The treated water (such as that produced by the process outlined in paragraph 17) is further subjected to treatment with a cation exchange resin in the proton form to remove any excess of Ca or Mg in the water from the settling basin and replace it with hydrogen ions. The resulting solution is then fundamentally what is required of the carbon regeneration step (exchange out the contaminant and add the promoter acid).

16. In the process outlined in paragraph 18 above, the spent cation exchange resin from the process in paragraph 18 is treated with a protonic acid, such as HCl, to regenerate the resin in the proton form and produce a soluble Ca or Mg salt.

17. The process may include the addition of the Ca and Mg brine from the resin regenerating unit to the settling tank (as in paragraph 17c. above) to aid the precipitation of the insoluble Ca or Mg salt of the contaminant.

18. The process may include the addition of acid or salt to the recycled water from the resin treatment step as needed to increase concentrations to those required for contaminant removal and promotion of sorbent activity.

19. One alternative to the water treatment step outlined in paragraphs 13-18 is to dispose of a portion of the entire aqueous contaminant stream by addition to a lime scrubber for the flue gas if one is in operation at the plant.

20. Another alternative to the water treatment step outlined in paragraphs 13-18, a small amount of water, referred to as blowdown, is disposed of.

If the acid added in the process outlined in paragraph 1 is an organic acid, or if some other acid with a pKa value of around 3-6, the addition of waste water containing this acid to the scrubber may derive a benefit by providing the buffer capability desired for the scrubber slurry.

### **Description of the Invention**

#### **FIELD OF THE INVENTION**

The invention relates to the process for the removal of mercury and other pollutant species from flue gas streams generated during the burning of fossil fuels, such as in a coal-fired utility. This removal process would utilize a sorbent to remove pollutants from the flue gas stream, which is at the same time interacting with other flue gas components that result in



poisoning or deactivating the sorbent and reducing its capacity or reaction rates with the pollutant species. The present invention provides a cost effective way to capture the pollutants by utilizing a process to regenerate the reactivity of the sorbent toward the pollutants or to regenerate the sorption capacity of the sorbent.

## **BACKGROUND OF THE INVENTION**

Burning fossil fuels generates a flue gas that contains mercury (and other trace elements) that was in the coal, plus oxides of sulfur and nitrogen (acid gas emissions) and particulates whose release to the environment must be controlled by use of sorbents, scrubbers, filters, precipitators, and other removal technologies. While other elements and/or flue gas pollutants may apply, the discussion provided is focused on mercury. Initially, mercury is released in combustion in the elemental form, but downstream of the boiler, in the ducts and stack of the combustion system, part of the elemental mercury is oxidized. The amount oxidized depends on the amount of HCl and other gases present in the flue gas. Amounts of mercury vary with the coal, but a typical concentration of mercury in the stream of combustion gas is about 5 parts per billion (ppb). Up to a pound of mercury per day may be emitted in some utilities.

Several types of mercury control methods for flue gas have been investigated, including injection of fine sorbent particles into a flue gas duct and passing the flue gas through a sorbent bed.

### ***Sorbent Injection Before Particulate Removal***

Fine particle injection sorbents include activated carbon, metal oxide sorbent, sodium sulfide particles (for example, see U.S. Pat. No. 6,214,304), basic silicate or oxide sorbents (for example, see the Ghorishi and Lancia references). When carbon particle injection is employed, the mercury chemisorbed to the sorbent particle is removed from the gas stream in a bag house or ESP and collected along with ash particulates (for example, see U.S. Pat. Nos. 4,889,698, 4,956,162, 5,672,323, 5,827,352, 6,027,551, 5,505,766. ). The disadvantage in these systems is that sorbent is used only once. The sorbents are relatively expensive and cannot be easily removed from the ash, regenerated and reused. These techniques also create solid waste disposal problems, and the spent sorbent may contaminate the collected ash for use in various applications.

### ***Sorbent Beds With Particulate Removal***

Carbon beds have also been utilized for mercury capture in flue gas. The German patent 34 26 059 describes the use of a very thick carbon bed for treatment of flue gases containing polyhalogenated compounds. Although the system employed at Dusseldorf would also have the potential for Hg removal (for example, see the Streng reference), it is not very practical to use such a thick bed. Spent sorbent is burned and not regenerated.



The GE-Mitsui-BF system employs a recirculating carbon bed, where mercury is removed along with acid gases (as ammonium salts) and the carbon is regenerated at high temperatures where ammonium sulfate is decomposed to SO<sub>2</sub> and N<sub>2</sub> and mercury is converted to the elemental form, which desorbs from the sorbent. Attrition of the sorbent results in a significant sorbent cost.

#### ***Sorbent Injection After Particulate Removal***

The process of injecting manganese oxide sorbent particles is described in US Patent Application 2002/0150516. Regeneration is claimed by removal of spent oxide particles from the reaction zone and rinsing with dilute aqueous acid.

#### ***Sorbent Beds After Particulate Removal***

Of particular interest in designing a mercury control process is to use the sorbent downstream of a particulate control device so the sorbent is not highly diluted with the ash particles. The sorbent could then be more easily regenerated and recycled. The prior art teaches several examples of this type of configuration and sorbent processing.

U.S. Pat. No. 5,607,496 teaches the oxidation of mercury on a metal oxide sorbent bed and subsequent absorption to sorbent. The sorbent bed follows the particulate removal equipment and thus the gas still contains the SO<sub>x</sub> and NO<sub>x</sub>, which react with the metal oxide sorbent to form the metal sulfates, that poison the bed. High temperatures are proposed to regenerate the bed. However, Hg is only partially removed from the sorbent at temperatures up to 500 degrees C. The sorbents do not work effectively after regeneration. The problem may be that manganese sulfate formed during the sorption cycle does not completely decompose back to an active manganese oxide form.

US Patent 6,383,981 describes a fixed MnO<sub>2</sub> or Fe<sub>2</sub>O<sub>3</sub> bed for removal of Hg from a hydrocarbon stream. No regeneration method is included.

A recent US Patent application (2001/0003116) describes the regeneration of a plate or honeycomb material composed of transition metal oxides that was used for sorption of mercury in flue gas. The claimed process involves heating the sorbent in a reducing gas stream to remove poisons, followed by impregnation with a polyfunctional complex-forming reagent containing the catalyst active component to restore mercury capture capacity. The first of these steps can remove the mercury, but it is unclear whether it actually removes the sulfate poison. The second of these steps is rather expensive, because one is actually reconstituting the reagent on the sorbent..

Porous beds containing a mercury oxidizing reagent on a solid support are described in several patents for removal of mercury from gas streams. These include peroxomonosulfate

(for example, see US Patent 4,786,483), triiodide or other mixed halogens (for example, see US Patents 3,194,629; 3,662,523), or sulfur (for example, see US Patents 3,194,629; 4,101,631, 4,708,853, 6,258,334). The reagent material is destroyed either by reaction with the flue gas during sorption or by attempts to regenerate the sorbent, there is no way to regenerate these sorbents, except by reimpregnation of the expensive reagent.

Amalgamating noble metals (gold, silver) on a suitable support can be regenerated by microwave heating (for example, see US Patent 6,136,072), but are expensive and not especially active for sorption in flue gas.

Following is a list of various background references:

Ghorishi, B.; Gullet, B.K. *Waste Manage Res.* 1993, 16, 582.  
 Lancia, A.; Musmarra, D.; Pepe, F.; Volpicelli, G. *Combust. Sci. & Technol.* 1993, 93, 277.  
 Streng, S.; Kassebohm, B. *Fuel Proc. Technol.* 1994, 39, 431.  
 US Patent 6,214,304 Rosenthal  
 US Patent 4,889,698 Moller  
 US Patent 4,956,162 Smith  
 US Patent 5,672,323 Bhat.  
 US Patent 5,827,352 Altman  
 US Patent 6,027,551 Hwang  
 US Patent 5,505,766 Chang  
 US Patent 5,607,496 Brooks  
 US Patent 6,383,981 Blankenship  
 US Patent 4,786,483 Audeh  
 US Patent 3,194,629 Dreibelbis, et al  
 US Patent 4,101,631 Ambrsini, et al  
 US Patent 3,662,523 Revoir  
 US Patent 4,708,853 Matviya  
 US Patent 6,258,334 Gadkaree  
 US Patent 6,136,072 Sjostrom  
 US Patent Application 2002/0150516  
 US Patent Application 2001/0003116 Neufert

## SUMMARY OF THE INVENTION

One feature of the invention is to provide a process for regenerating a spent sorbent which overcomes the above mentioned disadvantages of the prior art methods of this general type, and in which the regeneration effect is improved.

With the foregoing and other features in view there is provided, in accordance with the invention, a process for regenerating sorbents. The process includes washing the sorbent with agents selected to remove the anions that collect on the carbon during use in a flue gas and which poison the binding sites for mercury or other contaminants, and secondly to

impregnate the sorbent with agents that promote the chemisorption of mercury and other contaminants in the next service cycle of the sorbent.

This is achieved by the fact that, following or during the removal of the sulfate or other contaminants, the sorbent is washed with a reagent that restores mercury or other contaminant sorption capacity to the sorbent.

The invention applies to regeneration of a sorbent in a powdered form that has been injected into a flue gas stream after particulates have been removed, and have been collected on a suitable filtration or electrostatic precipitation (ESP) device. Examples of the composition of the sorbent are powdered activated carbon or a metal oxide or metal oxides, or a precious metal, or a composite of any of these. Alternatively, the sorbent is a bed material consisting of particles or fibers containing the compositions listed in the previous sentence. In both types, the poisoning contaminants from the flue gas are removed and an inexpensive promoting agent added to restore mercury sorption activity. This process of promoting the activity of the carbon itself contrasts with the earlier more expensive art of adding a reagent (such as peroxide, gold, triiodide, etc.) to the sorbent.

Although the invention is and described herein as a process for regenerating a sorbent, it is nevertheless not intended to be limited to the details described, since various modifications and structural changes may be made therein without departing from the spirit of the invention and within the scope and range of equivalents of the claims.

The construction and method of operation of the invention, however, together with additional objects and advantages thereof will be best understood from the following description of specific exemplary embodiments.

## DESCRIPTION OF EXEMPLARY EMBODIMENTS

Reactions between acidic components generated from flue gas poison the basic binding sites on the sorbent for oxidized mercury, which is a Lewis acid. Binding of other metal ions, such as boron, tin, arsenic, gallium Sb, Pb, Bi, Cd, Ag, Cu, Zn, or other contaminants that may also be Lewis acids, would suffer from loss of the intrinsic carbon basic sites.

To achieve the regeneration effect, it is necessary to remove the species that occupy the basic binding sites on the sorbent. These acid species far outnumber the bound mercury ions. The most serious competitor for the sites is sulfuric acid, formed from the SO<sub>2</sub> and SO<sub>3</sub> in the flue gas. The sulfate formed by reaction of the sulfuric acid with the basic sorbent site can be removed from the sorbent by washing with aqueous HCl or excess of chloride ion to exchange the sulfate or hydrogen sulfate ions. Other acids or reagents will also exchange these ions. Washing with an acid is more effective than washing with water since the anions exchange for the sulfate bound to the sorbent surface.



Washing with HCl and other acids also has the effect of promoting the subsequent oxidation of elementary mercury when the sorbent is returned to service. Adding the proton from the acid to a basic carbene site on the carbon edge structure forms a carbocation that accept electrons from the neutral mercury atom forming the oxidized mercury species that is bound to the sorbent surface.

The slurry of sorbent particles is subjected to a dewatering step using centrifugal force to remove the sorbent particles from the aqueous acid. Finally the sorbent is dried via a mild thermal treatment before returning to service. There are several reasons for dewatering the sorbent. By improving flow characteristics, this step will make the powdered or moving particle sorbent easier to handle during duct injection or in a contactor unit. Moisture may also block the pores preventing gas phase diffusion of elemental Hg, or may promote the sorption and subsequent oxidation of SO<sub>2</sub> to sulfuric acid.

The exemplary process also provides a method needed for effective treatment of the water used in the regeneration step. This water contains large amounts of sulfuric acid or sulfate salts if salts are used in the sorbent regeneration treatment. Other similar contaminants and a small amount of HCl or chloride salt will also be present. The waste water could be treated with lime or other Ca or Mg salts to precipitate sulfate, that is removed in a settling tank. The effluent water from the tank may contain a small amount of Ca or Mg. This water could be further treated with an H<sup>+</sup> form of a cation exchange resin to form desired acids such as HCl in the water, which is returned to the carbon regeneration unit. Additional acid could be added as needed to achieve the desired concentration. The resin is regenerated in turn to the H<sup>+</sup> form by cycling with HCl or other acid, and the waste solution from this step which contains CaCl<sub>2</sub> and MgCl<sub>2</sub> will simply added back to the sulfate removal unit.

Alternatively, the waste water solution from the dewatering step could be disposed of by adding to a scrubber slurry if one is available. This will convert the contaminant to insoluble Ca salts as scrubbers are supposed to do. The excess of exchanging agent can be useful in buffering the scrubber slurry as in the current art of scrubber technology.

Although the process for regenerating a deactivated sorbent is equally suitable for all forms of sorbent, an exemplary embodiment for a regeneration process according to the invention is described here.

### ***Carbon Sorbent***

A bench scale procedure based on the above description was to test the regeneration of several carbon sorbents, including the powdered Norit FGD sorbent, an aerogel carbon film sorbent, and a carbon filter consisting of fine carbon particles imbedded on a polymer fiber matrix.

The carbons were initially tested in a heated bed where a synthetic flue gas stream containing elemental mercury ( $11 \mu\text{g}/\text{m}^3$ ) was passed through the bed. Concentrations of total and elemental Hg in the effluent gas were determined using a Sir Galahad mercury CEM. The powdered sorbent was supported on a quartz filter during the test, and the other sorbents were tested as a triple layer.

A comparison of the original FGD powdered carbon sorbent with the sorbent after it is regenerated by washing with 0.1 N HBr, collecting the powder by centrifugation, and drying, revealed that the mercury capture activity is increased from an initial capture efficiency of about 50% of the Hg in the inlet to 100% capture. A comparison of the sorbent after it is regenerated again with HBr after its second service cycle revealed that the mercury capture is again 100% at the start of the test, and stays at a higher capture efficiency for a substantial number of minutes longer. Similar results were obtained with the carbon film and carbon fiber sorbents by regeneration with 0.1 N HCl.

FIG. 1 is a block diagram illustrating one example of the application of the sorbent regeneration technology of the present invention. FIG. 1 should not be considered limiting, but rather depicts a single method for incorporating the technology.

The process illustrated in FIG. 1 is laid out around the sorbent regeneration and treatment process 10 that, as a minimum, contacts the sorbent materials with the aqueous chemical reagent. This step could also include stronger agitation or grinding balls to expose fresh surfaces. Fresh activated carbon and recycled spent carbon are fed from separate storage silos 12 and 14 and metered at rates dependent upon mercury control feedback from the power system.

After the sorbent regeneration and treatment process 10, the resulting slurry is passed through a rough dewatering process 16 (e.g. hydroclone, vacuum filter, or other). The dewatering process 16 is followed by a final drying process 18 that utilizes hot process gas to bring the regenerated sorbent to a point of apparent dryness to allow subsequent handling and injection into the power system for mercury control. The majority of the aqueous solution from the dewatering process 16 is sent back to the regeneration reactor (via the path shown by arrow 20) along with a make up stream 22 comprised of water and fresh chemical reagent. A portion 24 of the aqueous solution is also sent to a system 26 that uses lime addition in a settling tank 28 and ion exchange (via ion exchanger 30) to remove sulfates prior to reuse. For some applications, the economics may favor replacing the sulfate removal step with a simple blow down stream and increased use of fresh aqueous chemical reagent.

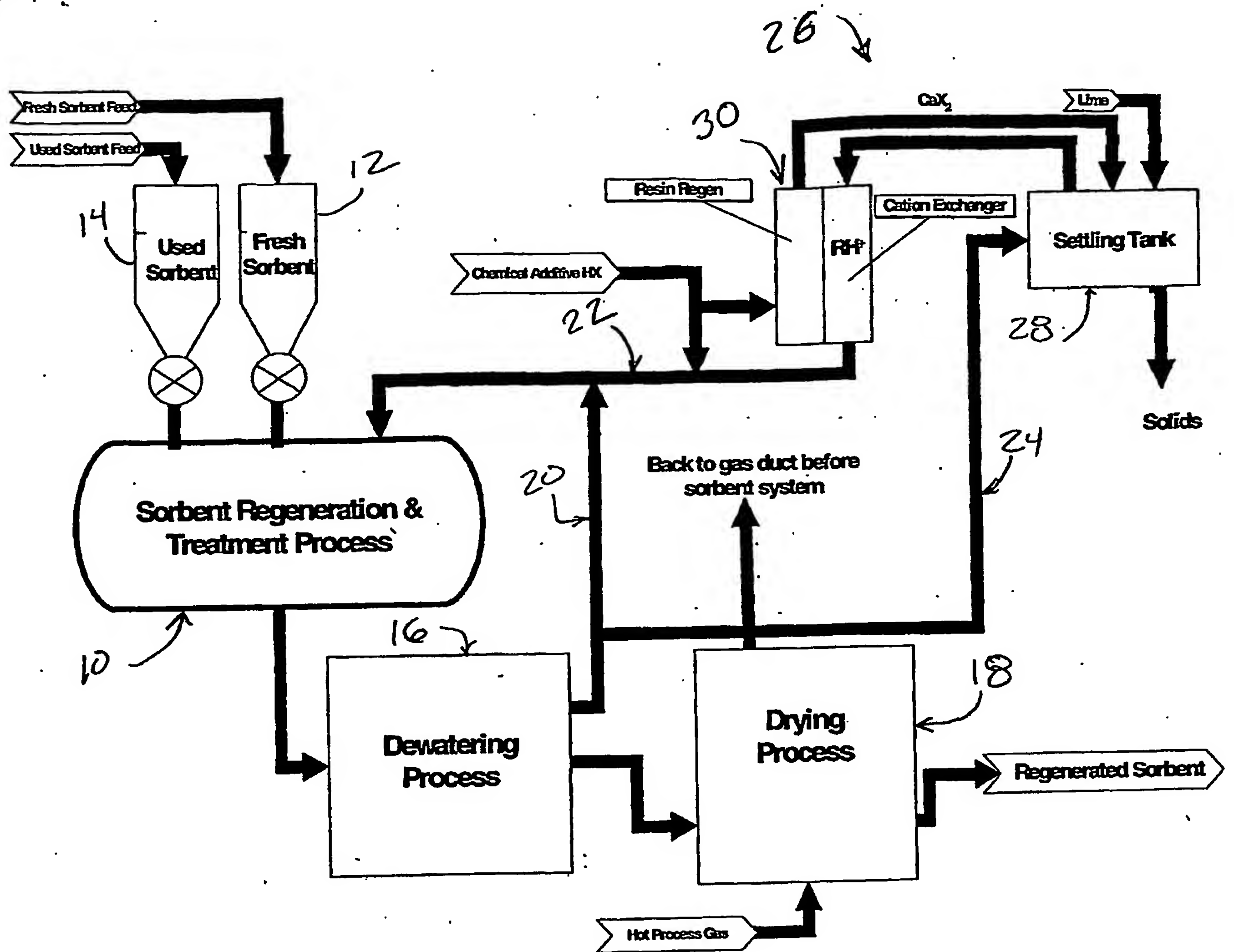


FIG. 1



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